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MATERIAL, AND MOLDED PRODUCT

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ABSTRACT:

PROBLEM TO BE SOLVED: To obtain a thermosetting resin composition which is very stable at an ordinary temperature and can be rapidly cured by heating at low temperatures by mixing a phenol/aldehyde precondensate with specified urea and/or thiourea curing agents.

SOLUTION: The curing agent is a urea reaction product obtained by mixing 1mol of urea and/or thiourea with 2.5-6mol of formaldehyde, adding an alkaline catalyst to the mixture to make it alkaline, reacting the entire by heating, adding an acidic catalyst to the reaction mixture when the pH of the reaction system is lowered to 7 or below, and reacting the entire mixture by heating on the acidic pH side. The resin composition mainly consists of a mixture comprising an aldehyde-curing precondensate comprising a precondensate of a monohydric phenol and/or a polyhydric phenol with an aldehyde and the above

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the woody material and moldings using the curing agent for initial condensates useful in an application of the binder used in case the moldings of fiber, or a wood chip or inorganic powder is manufactured, a nonwoven fabric, a fiber knit fabric, paper, etc., an impregnant, or the adhesives of wood, a thermosetting resin constituent, its hardening technique, and this thermosetting resin constituent.

[0002]

[Description of the Prior Art] As an application of the water-soluble binder used in case the moldings of fiber, or a wood chip or inorganic powder is manufactured conventionally, a nonwoven fabric, a fiber knit fabric, paper, etc., an impregnant, or adhesives of wood, 1 ** phenol, the polyhydric phenol, and an initial copolycondensation object of an aldehyde system like the initial condensate of a resol type phenolaldehyde system or the initial copolycondensation object of a phenol resorcinol aldehyde system are used.

[0003]

[Problem(s) to be Solved by the Invention] However, when a material lacking in thermal resistance, such as a fiber and wood chip, is used according to [there is a fault which must save at low temperature since the initial condensate of the above-mentioned resol type phenolaldehyde system is unstable at a room temperature, and is said that the term which can be saved is very short and moreover the 180-200-degree C heating at high temperature is usually required for the hardening, and] such a heating at high temperature, heat deterioration is produced, and a heat energy cost also becomes high. On the other hand, in the case of 1 ** phenol, the polyhydric phenol, and an initial copolycondensation object of an aldehyde system like the initial copolycondensation object of a phenol resorcinol aldehyde system, when it is necessary to add an aldehyde and it uses formalin and an aldehyde like a paraformaldehyde as a curing agent as a curing agent in the case of use, since reactivity is too high, the pot life in the room temperature after curing agent addition is short, a working life is it 2 - 3 hours that it is long, and there is a fault that workability is very moreover, even when using a decomposed type hexamethylenetetramine instead of formalin and a paraformaldehyde as a curing agent Mix this 1 ** phenol, a polyhydric phenol, and the initial copolycondensation object of an aldehyde system as a binder, or Decomposition of a hexamethylenetetramine happens in the phase of predrying after carrying out application sinking in as an application or an impregnant, and hardening of this first time copolycondensation object advances considerably. Furthermore, when hardening of this first time copolycondensation object advanced quickly at the room temperature, and this predrying object lost the moldability and could not save it for a long period of time, it had a **** trouble. In order to improve the pot life after curing agent addition of a resorcinol system resin, amino resins, such as a methylolurea, a formaldehyde resin, a methylation methylolurea, a methylol melamine, a melamine formaldehyde resin, and a methylation methylol melamine, are added instead of an aldehyde as a curing agent, and the hardening technique which carries out heating hardening or more by pH seven is indicated (JP,57-053536,A). However, there was a trouble where it could not be used since the amino resin which heating of 180 degrees C or more is needed for hardening, and cannot attain the purpose told [hardening more and] to a low-temperature short time, and was added in the pH seven or less acidity side will cause a separation, the freezing, etc. although the working life after curing agent addition is extended if this technique is applied to above-mentioned 1 ** phenol, polyhydric phenol, and initial copolycondensation object of an aldehyde system.

[0004]

[Means for Solving the Problem] the curing agent which is a resultant (one mol of the urea and/or thiourea which hold a compatibility as the water solubility also with a good acidity side as a means for this invention solving the above-mentioned conventional technical problem, and 2.5-6 mols of formaldehyde) -- providing -- further -- the moldings which used the hardening technique of the thermosetting resin constituent which makes a principal component mixture of the initial condensate of 1 ** phenol and/or a polyhydric phenol, and an aldehyde and this curing agent, and this thermosetting resin constituent, and this thermosetting resin constituent is offered

[0005] this invention is explained in detail below. The thermosetting resin constituent of this invention makes a subject mixture with the curing agent which are an initial condensate (henceforth initial condensate A) of an aldehyde hardening type like the initial condensate of 1 ** phenolaldehyde system, a polyhydric phenol and the initial condensate of an aldehyde system, and/or 1 ** phenol, a polyhydric phenol and the initial condensate of an aldehyde system, and urea system resultant B.

If 1 ** phenol used for initial condensate A of [** 1 Phenol] this invention is illustrated Alkylphenols, such as a phenol, o-cresol, m-cresol, p-cresol, ethylphenol, iso-propyl phenol, a xyleneol, 3, 5-xyleneol, butylphenol, t-butylphenol, and a nonyl phenol, o-fluorophenol, m-fluorophenol, p-fluorophenol, o-chlorophenol, m-chlorophenol, p-chlorophenol, o-***** phenol, m-***** phenol, p-***** phenol, o-iodine phenol, m-iodine phenol, ortho aminophenol, m-aminophenol, para aminophenol, ortho nitrophenol, m-nitrophenol, p-nitrophenol, a 2, 4-dinitrophenol, 2, 4, They are independent or two or more sorts of mixture of 1 ** phenols, such as 1 ** phenol substitution products, such as 6-trinitrophenol, and a naphthol.

[0006] If the polyhydric phenol used for initial condensate A of a [polyhydric-phenol] this invention is illustrated, they are independent or two or more sorts of mixture of polyhydric phenols, such as gold [a cull / a resorcinol, an alkyl resorcinol, pyrogallol, a catechol, an alkyl catechol, hydroquinone alkyl hydroquinone, a phloroglucine, a bisphenol, dihydroxy naphthalene, and], a urushiol, *****, a ***** call, a register Norian, *****, tannin, pyrogallol tannin, catechol tannin, adding-water resolvability tannin, and condensed type A desirable thing is a resorcinol or an alkyl resorcinol among these polyhydric phenols, and especially a desirable thing is an alkyl resorcinol with the reaction rate quicker than a resorcinol with an aldehyde. As the above-mentioned alkyl resorcinol For example, 5-methyl resorcinol, 5-ethyl resorcinol, 5-propyl resorcinol, a 5-n-butyl resorcinol, 4, 5-dimethyl resorcinol, 2, 5-dimethyl resorcinol, 4, 5-diethyl resorcinol, 2, 5-diethyl resorcinol, 4, 5-dipropyl resorcinol, There are 2, 5-dipropyl resorcinol, a 4-methyl-5-ethyl resorcinol, a 2-methyl-5-ethyl resorcinol, a 2-methyl-5-propyl resorcinol, 2 and 4, a 5-trimethyl resorcinol, 2 and 4, a 5-triethyl resorcinol, etc. Since the polyhydric-phenol mixture obtained by dry distillation of the Estonia ** oil shale contains various reactant high alkyl resorcinols besides 5 ***** resorcinol so much cheaply, it is the most desirable polyhydric-phenol raw material in this invention.

[0007] In order to manufacture initial condensate A of a [aldehyde and/or aldehyde donator] this invention, the above-mentioned 1 ** phenol and/or a polyhydric phenol, an aldehyde, and/or an aldehyde donator are condensed. The above-mentioned aldehyde donator means the compound which carries out generation supply of the aldehyde, if it decomposes. If the above-mentioned aldehyde and an aldehyde donator are illustrated, they are independent or two or more sorts of mixture, such as formalin, formaldehyde, a paraformaldehyde, a trioxane, an acetaldehyde, a propionaldehyde, a polyoxymethylene, a trichloroacetic aldehyde, a hexamethylenetetramine, a furfural, glyoxal, n-butyraldehyde, a caproaldehyde, an allyl-compound aldehyde, the Benz aldehyde, a crotonaldehyde, an acrolein, a tetrapod oxy-methylene, a phenylacetaldehyde, o-torr aldehyde, and a salicylaldehyde.

[0008] In a [condensation catalyst] this invention, in the case of condensation with 1 ** phenol and/or a polyhydric phenol, an aldehyde, and/or an aldehyde donator, if required, an acid or alkaline condensation catalyst will be added. Usually let the addition be an amount below 6 % of the weight (for it to only consider as % below) to 1 ** phenol and/or a polyhydric phenol. As the above-mentioned acid catalyst, for example A hydrochloric acid, a sulfuric acid, a phosphoric acid, a boric acid, oxalic acid, formic acid, An acetic acid, a butyric acid, a benzenesulfonic acid, a phenolsulfonic acid, Para toluenesulfonic acid, Inorganic or organic acids, such as a naphthalene-alpha-sulfonic acid and a naphthalene-beta-sulfonic acid, Or acid anhydrides, such as ester of organic acids, such as oxalic acid dimethyl ester, and a maleic-acid anhydride, Manganese hydroxide, chromium hydroxide, an iron hydroxide, hydroxylation lead, copper hydroxide, zinc acetate, Formic-acid zinc, benzoic-acid zinc, a zinc chloride, ferric chloride, manganese acetate, a cadmium acetate, An ammonium chloride, an ammonium sulfate, an ammonium nitrate, an ammonium oxalate, An ammonium acetate, ammonium phosphate, an ammonium thiocyanate, An ammonium salt, monochloroacetic acid, and its specific salts, such as imido sulfonic-acid ammonium, There are urea adducts, such as a hydrochloride of aminess, such as organic halogenides, such as alpha and alpha' dichlorohydrin, a triethanolamine hydrochloride, and an aniline hydrochloride, a salicylic acid urea adduct, a stearin acid urea adduct, and an oenanthic-acid urea adduct, an N-trimethyl taurine, etc. As the above-mentioned alkaline catalyst, aminess, such as the weak-acid salts of alkali metal, such as the hydroxide of metals, such as a sodium hydroxide, a potassium hydroxide, a barium hydroxide, a calcium hydroxide, a calcium oxide, a barium oxide, a magnesium oxide, a zinc oxide, cobalt oxide, a lead oxide, nickel oxide, and an aluminum oxide, an oxide, a sodium carbonate, a sodium sulfite, sodium acetate, and a sodium phosphate, ammonia, a trimethylamine, a triethanolamine, a hexamethylenetetramine, and a pyridine, are illustrated, for example. You may use together the above-mentioned acidity or two or more sorts of alkaline catalysts.

[0009] Although the indifferent water is used as a solvent of initial condensate A in a [solvent] this invention If required, further A methanol, ethanol, an isopropanol, n-propanol, an isopropanol, n-butanol, an isobutanol, sec-butanol, t-butanol, n-amyl alcohol, isoamyl alcohol, n-hexanol, A methyl amyl alcohol, 2-***** Norian, n-heptanol, n-octanol, trimethyl nonyl alcohol, a cyclohexanol, benzyl alcohol, furfuryl alcohol, a tetrahydrofurfuryl alcohol, loon ethyl alcohol, Alcohols, such as diacetone alcohol, An acetone, a methyl acetone, a methyl ethyl ketone, a methyl-n-propyl ketone, a methyl-n-butyl ketone, a methyl isobutyl ketone, a diethyl ketone, a G n-propyl ketone, a diisobutyl ketone, an acetonylacetone, a methyl oxide, a cyclohexanone, Ketones, such as a methylcyclohexanone, an acetophenone, and camphor Glycols, such as ethylene glycol, a diethylene glycol, a triethylene glycol, a propylene glycol, a trimethylene glycol, and a polyethylene glycol Glycol ethers, such as an ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, an ethylene glycol isopropyl ether, the diethylene-glycol monomethyl ether, and the triethylene-glycol monomethyl ether The ester, the derivatives of those of the above-mentioned glycols, such as an ethylene glycol diacetate and diethylene-glycol-monoethyl-ether acetate, The addition use of independent or two or more sorts of mixture of water fusibility organic solvents, such as ether, such as 1 and 4-dioxane,

a diethyl cellosolve, a diethyl carbitol, ethyllactate, isopropyl lactate, a diethylene glycol diacetate, and a dimethylformamide, can be carried out. While an acetone etc. is a solvent, when using an alkyl resorcinol, it acts also as the complexing agent, and it brings a quieter reaction.

[0010] Without making the bottom of acidity or alkaline condensation catalyst presence, or a condensation catalyst exist, initial condensate A of a [initial condensate A] this invention is under heating, or in ordinary temperature, the condensation reaction of the above-mentioned 1 ** phenol and/or a polyhydric phenol, an aldehyde, and/or the aldehyde donator is carried out, and they are obtained. The addition of the aldehyde in this case and/or an aldehyde donator is usually 0.2-5 mols to one mol of 1 ** phenols, and is usually about 0.1-0.8 mols to a polyhydric phenol. When carrying out the copolycondensation of 1 ** phenol and the polyhydric phenol, it is desirable to usually carry out a polyhydric phenol in amount of about 0.1-2 mols to one mol of 1 ** phenols. Or he has no presence of a catalyst. the copolycondensation of 1 ** phenol, a polyhydric phenol, an aldehyde, and/or the aldehyde donator is carried out -- making -- the bottom of presence of alkali or an acid catalyst -- How to condense simultaneously 1 ** phenol, a polyhydric phenol, an aldehyde, and/or an aldehyde donator, 1 ** phenol and/or the initial condensate of 1 ** phenolaldehyde -- a polyhydric phenol -- and -- or, although arbitrary technique, such as the technique of adding and carrying out the copolycondensation of an aldehyde and/or the aldehyde donator to a polyhydric phenol and the initial condensate of an aldehyde if required, may be adopted Desirable technique is made into the initial condensate of a resol type phenolaldehyde which condenses 1 ** phenol, an aldehyde, and/or an aldehyde donator under alkali catalyst presence, and has a methylol machine. Subsequently, a polyhydric phenol and/or a polyhydric phenol, and the initial condensate of an aldehyde system are added to this condensate, and if required, it is the technique of adding and carrying out the copolycondensation of an aldehyde and/or the aldehyde donator. In the case of the above-mentioned copolycondensation, it is usually added to one mol of 1 ** phenols at a rate of 0.2-6.0 mols of aldehydes, and 0.1-2.0 mols of polyhydric phenols. As a third component of the above-mentioned initial condensate A a request -- if -- a urea, a melamine, thiourea, benzoguanamine, toluene, a xylene, a cumarone, a cyclohexanone, cashew oil, tannin, *****, a shellac, rosin or a rosin derivative, a petroleum resin, a methanol, ethanol, and an isopropanol -- n-butanol, an isobutanol, ethylene glycol, a diethylene glycol, a polyethylene glycol, a glycerol, furfuryl alcohol, the linseed oil, tung oil, castor oil, Kinds, such as an acetone and a methyl ethyl ketone, or two sorts or more are made into a copolycondensation agent, a modifier, or a complexing agent, and even if it adds and it denaturalizes a copolycondensation or by mixing among a reaction or after a reaction end at the time of reaction start of condensation or a copolycondensation, it does not interfere. Although the aqueous solution of the above-mentioned initial condensate A is usually stable in the with a pH of eight to nine or more alkalinity side, it is unstable at a neutrality or acidity side, and the problem which causes a layer separation arises. For solving this trouble, a sulfo methyl group and/or a ***** methyl group are introduced into this first time condensate A, and they are a sulfo methylation object and/. Or considering as a ***** methylation object is desirable. The water solubility in large pH field reached [from the alkalinity of this first time condensate A] acid is remarkably improved by introduction of the above-mentioned sulfo methyl group and/or a ***** methyl group, and the thermosetting resin constituent of a stable this invention is obtained.

[0011] In a ***** methylation agent, the addition reaction of the ** which also spreads a sulfo methylation agent and/or after a reaction end in the middle of the time of this condensation-reaction start or a reaction is carried out, and the sulfo methylation object and/or ***** methylation object of initial condensate A of the [sulfo methylation object and ***** methylation object] above-mentioned phenolaldehyde system are obtained [**], in case 1 ** phenol and/or a polyhydric phenol, an aldehyde, and/or an aldehyde donator are made to condense. since it is desirable to add in case the above-mentioned sulfo methylation agent and/or a ***** methylation agent condense 1 ** phenol, an aldehyde, and/or an aldehyde donator under alkali catalyst presence and it considers as the initial condensate of a phenolaldehyde system and it methylates [sulfo-] and/or methylates [*****-] 1 ** phenol beforehand in this case -- an aldehyde and/or an aldehyde donator, and condensation -- or even if it carries out a copolycondensation, it does not interfere If the above-mentioned initial condensate A is sulfo-methylated and/or ***** methylated, the working life at the time of the reaction activity with the aldehyde of this first time condensate A falling, and adding an aldehyde and/or an aldehyde donator as a curing agent will be extended. Moreover, since the reaction activity with the aldehyde of this copolycondensation object will fall most if pH is kept general into fine acidity from neutrality although the water solubility of this first time condensate A increases and good water solubility comes to be shown in large pH field by the side of neutrality or acidity by introduction of a sulfo methyl group and/or a ***** methyl group, a working life becomes the longest. Since there is a possibility that hardening may be too slow even if it adds this urea system resultant that a fall of the reaction activity with an aldehyde may be remarkable, and carries out a postscript according to sulfo methylation and/or ***** methylation, when initial condensate A is the initial condensate of 1 ** phenol, it is desirable to choose 1 ** phenol, a polyhydric phenol, and the initial copolycondensation object of an aldehyde system as initial condensate A in this invention. In the above-mentioned sulfo methylation, it is desirable to make a sulfo methyl group into the domain of 0.05-0.5 mols to one mol of 1 ** phenols. When a sulfo methyl group is 0.05 mols or less, a fall of the reaction activity with an aldehyde is inadequate, and short ** past ***** has a working life. Moreover, if a sulfo methyl group is 0.5 mols or more, the reaction activity with an aldehyde will fall too much, the hardened material which has sufficient intensity by heating of about 120-140 degrees C is not obtained, and the fault that the durability of a hardened material is bad arises. It is desirable also in ***** methylation, to make a ***** methyl group into the domain of 0.05-0.5 mols to the monohydric phenol of one mol, since it is the same as that of the case of sulfo methylation. If initial condensate A is sulfo-methylated and/or ***** methylated, the stable mixed constituent with a urea system

resultant will be obtained also neutrality and under acid. That is, in pH field to which the reaction activity of the urea system resultant of this invention becomes higher, this mixed constituent can be used and low temperature and the advantage which may advance a hardening reaction by short-time heating arise more.

[0012] As a sulfo methylation agent used in a [sulfo methylation agent and ***** methylation agent] this invention The water-soluble sulfite with quaternary amines, such as a sulfurous acid, a pile sulfurous acid or a meta-pile sulfurous acid, alkali metal or a trimethylamine, and benzyl trimethylammonium, or the fourth class ammonium, Hydroxy alkane sulfonic acids, such as hydroxy methanesulfon acid chloride obtained by the reaction of these water-soluble sulfites and aldehydes, The hydroxyphenyl alkane sulfonate obtained from the initial condensate of a resol type phenolaldehyde and the above-mentioned sulfite is illustrated. As a ***** methylation agent used in this invention, alkyl sulfoxylate, such as the sodium hydrosulfites of alkali metal, such as aliphatic series, such as sodium formaldehyde sulfoxylate (Rongalite) and benzaldehyde sodium ***** , alkali-metal sulfoxylate of an aromatic aldehyde, a sodium sodium hydrosulfite, and magnesium hydro ***** , and alkaline earth metal and sodium ethyl sulfoxylate, is illustrated. Since a sulfo methylation reaction advances in parallel to a ***** methylation reaction when using a ** 2 thionic-acid alkali-metal salt as a ***** methylation agent, a sulfo methylation object generates with a ***** methylation object.

[0013] Urea system resultant B used as a curing agent in a [urea system resultant B] this invention is obtained by two staircase reactions to which a urea and/or thiourea, and formaldehyde are subsequently made to react by the acidity side by the alkali side at first. The addition of the formaldehyde to a urea and/or one mol of thiourea is far used so much rather than the case of a manufacture of the usual initial condensate of a urea system, and after the domain of 2.5-6 mols is suitable for it, it adds an alkaline catalyst and sets pH to pH 8-12 preferably eight or more, the pyrogenetic reaction of it is usually carried out at the temperature of 30-80 degrees C. The curing agent for the initial condensates of the aldehyde hardening type which will consist of the urea system resultant B of this invention if an acid catalyst is newly added when pH is [seven or less] desirable and it falls to pH 6-7, although pH of this reaction mixture fell in advance of a reaction, and it adjusts to pH 4-6 preferably pH six or less] and a 1 - 2 hours pyrogenetic reaction is usually carried out at 80-100 degrees C the appropriate back is obtained. The alkaline catalyst and acid catalyst which are added here have the condensation catalyst used when manufacturing the above-mentioned initial condensate A, and the same usable catalyst. Furthermore, in the generation reaction of urea system resultant B, it is the phase of making it reacting by the alkali side, for example, the buffer of potassium chloride, a sodium chloride, a boric acid, a borax, a sodium metaborate, 2 sodium borates, a sodium tetraborate, a sodium pentaborate, 6 sodium borates, 8 sodium borates, an ammonium chloride, a phosphoric acid, phosphoric-acid disodium, a glycine, dimethyl ***** sodium et al. may be added, and a fall of pH may be prevented. This urea system resultant B has the property which unlike the usual initial condensate of a urea system the solubility over water is very good, and does not dissociate and solidify in pH six or less acid field, but the transparent or translucent aqueous solution stable for a long period of time is obtained at a room temperature, and is stably mixed in the initial condensate A of an aldehyde hardening type like the initial condensate of a phenolaldehyde system, an acid, and large pH field of alkali. Furthermore, although it is checked that it is what the disengagement formalin content of this urea system resultant B usually reaches to about 5 - 20% of the weight according to the sodium sulfite method, therefore has the reactivity with this high urea system resultant B, even if ***** straw ***** urea system resultant B is very stable in ordinary temperature also to it and it adds to initial condensate A of an aldehyde hardening type, it does not gel in ordinary temperature for a long time. On the other hand, when the formaldehyde aqueous solution of equivalent concentration is added to initial condensate A of an aldehyde hardening type, it gels for a short time. Moreover, when the addition of the formaldehyde to a urea and/or one mol of thiourea is 2.5 mols or less, a stability and the miscibility with initial condensate A of an aldehyde hardening type deteriorate remarkably, and gel or separate a resultant from urea system resultant B of this invention in the time of mixture, or a short time after mixture. this urea system resultant B -- 100 degrees C or less as aqueous solution -- the bottom of an ordinary pressure or reduced pressure -- concentration and xeransis -- carrying out -- a paste and flakes -- or it is provided as powdered Although urea system resultant B of this invention is very stable, if it is heated at 100 degrees C or more, it will start an activation, as described above, in ordinary temperature, if this is further performed under acid, it will activate it much more actively, and it acts as a curing agent to initial condensate A of an aldehyde hardening type. And the activity to the hardening reaction has the property that it is more remarkable than different wave node ring compounds, such as ***** which has other ***** roll machines, an ethylene urea, a propylene urea, and a ***** system, or a methylolurea, a formaldehyde resin, a methylol melamine, a melamine formaldehyde resin, etc., and is high. Therefore, this urea system resultant serves as the very effective curing agent which carries out heating hardening of an initial condensate of an aldehyde hardening type like the initial condensate of a phenolaldehyde system in a low-temperature short time.

[0014] Although made to usually harden the initial condensate A of an aldehyde hardening type like the initial copolycondensation object of [curing catalyst] phenol resorcinol formaldehyde by the alkali side, this initial condensate A of an aldehyde hardening type can also make a neutrality or acidity side harden in this invention. For example, in the case of the thermosetting resin constituent of this invention which consists of initial condensate A and urea system resultant B into which the sulfo methyl group and/or the ***** methyl group are introduced, even if it adds a still acid or alkaline compound as a curing catalyst in the case of hardening in addition to the above-mentioned urea system resultant B, it does not interfere. If the above-mentioned acid curing catalyst is illustrated, a hydrochloric acid, a sulfuric acid, a nitric acid, a phosphoric acid, a pyrophosphoric acid, A polyphosphoric acid, a sulfonic acid, a hydrobromic acid, a hydroiodic acid, trichloroacetic acid,

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